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Measuring the Interaction Force Between a Tip and a Substrate Using a Quartz Tuning Fork Under Ambient Conditions

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Tuning forks mounted with sharp tips provide an alternate method to silicon microcantilevers for probing the tip-substrate interaction in scanning probe microscopy. The high quality factor and stable resonant frequency of the tuning fork allow accurate measurements of small shifts in the resonant frequency as the tip approaches the substrate. To permit an accurate measure of surface interaction forces, the electrical and piezomechanical properties of a tuning fork has been characterized using techniques derived from scanning probe microscopy. After proper calibration, representative interaction force data for a conventional Si tip and an HOPG substrate are obtained under ambient conditions.

Keywords: Scanning Probe, Quartz Tuning Fork, Interaction Force.

RESEARCH ARTICLE

1. INTRODUCTION

It is well known that quartz tuning forks can be used as force detectors for SPM. Originally designed for high precision frequency control,[1–4] quartz tuning forks are widely used in clocks, watches and other frequency standard. Most of the commercial tuning forks have a standard resonant frequency of 2^{15} Hz. They are robust and extremely stable in frequency compared to conventional AFM cantilevers. Their high mechanical quality factor, which is in the order of 10^4 in air, makes them sensitive to pN shear and normal forces. The piezoelectric effect of quartz crystal, which yields an electrical signal proportional to deformation, makes the measurement of the amplitude of oscillation very simple compared to an optical beam bounce measurement scheme. Due to these advantages over conventional cantilevers, tuning forks have been successfully used as force sensors in SPM, yielding atomic resolution images at low temperature and ultra high vacuum.[5]

In what follows, we will review a few of the unique properties of a quartz tuning fork and will describe a new method of calibrating the amplitude of oscillation of the tuning fork. Once the tuning fork is calibrated, reliable force versus distance data can be obtained under a variety of conditions. The results of a few of our initial studies under ambient conditions will be presented.

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2. EXPERIMENTAL DETAILS

2.1. Mechanical Properties of a Quartz Tuning Fork

As with any resonator, it is important to know important characteristics such as effective mass, spring constant, resonance frequency and quality factor to characterize a tuning fork force detector. The resonant frequency can be measured by sweeping the frequency of driving signal applied to the tuning fork electrode and measuring the resulting amplitude and phase of the current directly. From this data, the quality factor can be estimated in a straightforward way. The spring constant and the effective mass are more difficult to measure directly. However, these characteristics can be estimated from models based on their geometrical dimensions.[1,4] The effective mass and spring constant are given by.[1,6]

$$m_e = 0.2427\rho(Lwt)$$
$$k = \frac{1}{4}Yw(t/L)^3 \tag{1}$$

where L is the length of two prongs of tuning fork, w is the width of one prong, t is the thickness of the prongs, Y is the Young's modulus of quartz, which is 7.87×10^{10} N/m^2, and ρ is the mass density, which is 2.65×10^3 kg/m^3 for quartz. The resonance frequency of a tuning fork also can be calculated from its geometrical dimensions using.[1,6]

$$f_0 = 1.015\frac{t}{2\pi L^2}\sqrt{\frac{Y}{\rho}} \tag{2}$$

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The geometrical dimensions of the tuning fork used in this work are $L = 3.28$ mm; $t = 0.40$ mm; $w = 0.33$ mm. The calculated effective mass is 2.78×10^{-7} kg; the calculated spring constant is 11.78 kN/m; the calculated resonant frequency (f_0) is 32.73 kHz. The resonance frequency calculated using the geometrical dimensions is consistent with the measured resonance frequency to within a few percent.

2.2. The Quartz Tuning Fork as an SPM Cantilever

For a quartz tuning fork to be used in a scanning probe microscope, a sharp probe tip must be attached to the leg of the tuning fork. In our experiments, this was accomplished by attaching a commercially available silicon AFM microcantilever to the free end of one leg of a tuning fork.

For normal force detection, the silicon probe is oriented perpendicular to the arm of the tuning fork and the oscillation of the probe is also perpendicular to the substrate surface. The attachment of the silicon probe must be of high quality. The microcantilever must be in close contact with the tuning fork to avoid any spurious absorption of energy which will decrease the sensitivity of the force detection.

When received, the tuning fork is encased in an evacuated metal can. After carefully removing the can, the resonance can be measured under ambient conditions. The additional mass of the attached silicon tip and glue usually drops the resonant frequency about 50 ~ 100 Hz, and it also drops the quality factor as shown in Figure 1. An understanding of the shape of the resonance shown in Figure 1 requires a more complete understanding of the equivalent electrical circuit of this oscillator.

2.3. Electrical Properties of a Quartz Tuning Fork

In order to drive a quartz tuning fork into oscillation, either an AC voltage must be applied across the two electrodes or an external vibration must be used to physically shake the quartz tuning fork. Using either approach, it is necessary to understand the equivalent electrical circuit of the quartz tuning fork to understand the frequency dependence of the current generated by the resulting oscillation.

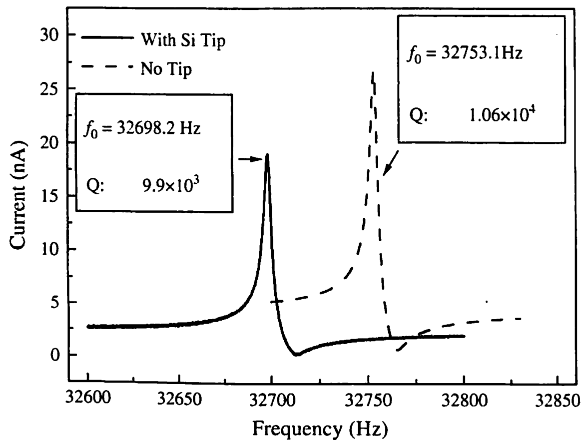

Fig. 1. The resonance frequency of a tuning fork before and after attaching a silicon microcantilever.

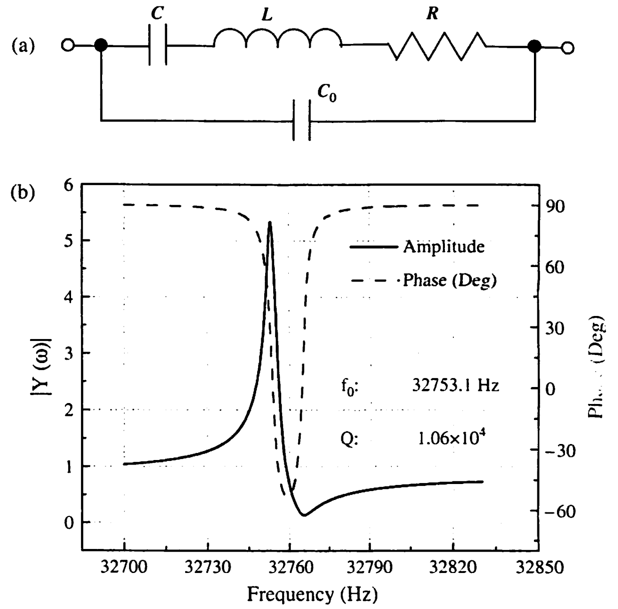

Fig. 2. (a) Electrical model of a quartz tuning fork. (b) Calculated frequency response for the admittance (both phase and amplitude) of a quartz tuning fork.

The electrical model, shown as Figure 2(a), can be used to understand the electrical properties of a quartz tuning fork.[7,8] The motional capacitance C, series resistance R and equivalent inductance L represents a resonator which a resonant frequency of $f_0 = 1/2\pi\sqrt{LC}$ and a damping constant of $\gamma = R/L$. The extra capacitor C_0 is required to account for any parasitic capacitance from electrical contacts and connected cables. The quartz tuning forks used in our experiments have a serial resistance of 35 kΩ, a motional capacitance of 3.5 fF, a parasitic capacitance of 1.6 nF and an equivalent inductance of 6.7 kH.[9]

When a tuning fork is driven at its resonant frequency, the current is maximized. When driven at a frequency away from its resonant frequency, the tuning fork's response is dominated by the parasitic capacitor C_0 which produces a parallel current I_p given by $I_p = j2\pi f C_0$. The current through the parasitic capacitance produces a minimum in the admittance located about 12 Hz above the resonant frequency and directly accounts for the lack of symmetry of the frequency response curve as shown in Figure 2(b).

To eliminate the asymmetry caused by the parasitic capacitance, a circuit was designed as shown in Figure 3(a). The driving signal of the tuning fork has 180° difference in phase with the voltage source applied to the compensating capacitor C. Adjusting the variable capacitor C to a proper value, the current through the variable capacitor can negate the current through the parasitic capacitance. Therefore, the current amplified by the current amplifier in

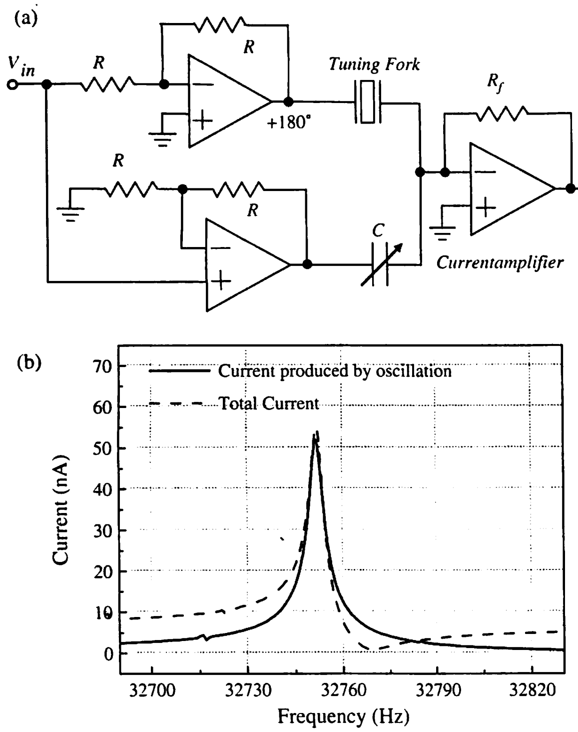

Fig. 3. (a) The detection circuit for a tuning fork force sensor to eliminate the asymmetry in the resonance curve. (b) Frequency response of current through a quartz tuning fork with a driving signal V_{in} of 10 mV. The solid curve shows the current fed into the current-voltage amplifier show in Figure 5(a). The current is only due to the osscillation of the quartz oscillator. The dashed curved shows the total current without the compensation circuit.

Figure 3(a) only contains a contribution from the oscillation of the tuning fork. The resulting measured current as a function of drive frequency is plotted in Figure 3(b) and clearly shows the symmetric nature of the frequency response curve of a quartz tuning fork when the effect of the parasitic capacitance is cancelled.

2.4. Measuring the Piezo Electromechanical Coupling Constant

From the data shown in Figure 3(b), the admittance as a function of frequency ω can be analyzed and the electrical parameters C, C_0, L and R can be determined. However, the oscillation amplitude of the tuning fork (in nanometers) still can not be determined even if the current flowing through the tuning fork is known. In order to calibrate the mechanical amplitude of the tuning fork, an additional parameter α (often called the piezo electromechanical coupling constant).[8] is required. The piezo electromechanical coupling constant, in units of Coulomb per meter (C/m), describes the charge induced on the piezo material for a given mechanical deflection. This constant is often obtained by measuring the current flowing through the tuning fork while the mechanical oscillating amplitude is measured using an optical interferometer.[7] This constant α is a characteristic parameter of a tuning fork oscillator and modifications in operating conditions, such as the attachment of a tip to the leg of the tuning fork, should not change this constant.[8]

With the piezo electromechanical coupling constant α, the mechanical oscillating amplitude $x(t)$ and the current $I(t)$ flowing through the resonator can be related as described elsewhere.[7] For a sinusoidal oscillation,

$$I_{\text{peak}} = \alpha \omega x_{\text{peak}} \tag{3}$$

where ω is angular frequency of the oscillation. The important result of Eq. (3) is that to determine α, the amplitude of the tuning fork must be accurately measured.

2.5. Calibration of the Oscillation Amplitude

In order to calibrate the tuning fork's amplitude of oscillation as a function of the current flowing from it requires a calibration of the piezo-electromechanical coupling constant. The common method to calibrate the oscillating amplitude of the tuning fork force sensor is utilizing optical interference. In our experiments, a new method is introduced using an AFM cantilever. The experimental set up is shown in Figure 4(a) where the leg of a tuning fork, acting as a substrate, was scanned by a well-calibrated AFM. After calibrating the photodetector output, a quartz

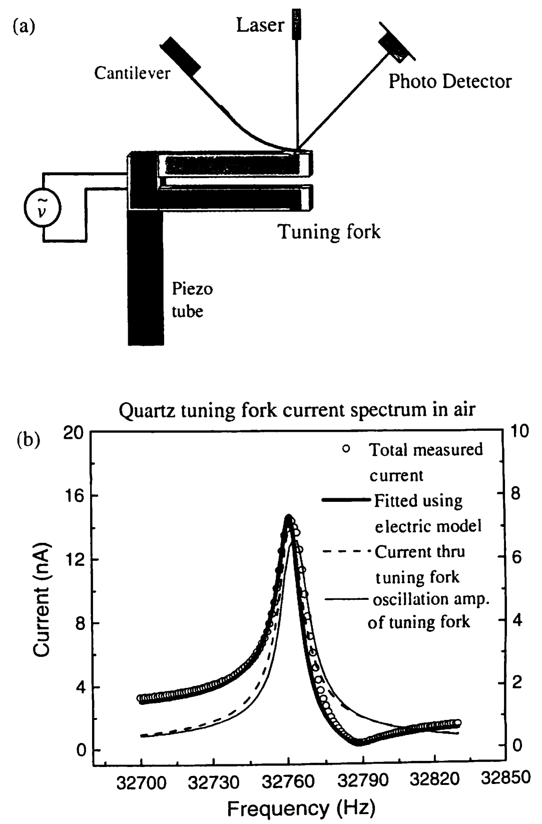

Fig. 4. (a) Schematic showing how the oscillating amplitude of a tuning fork was measured using an AFM cantilever. (b) The current response and the mechanical oscillation of a tuning fork.

tuning fork was excited by a 5 mV signal. The current response of the tuning fork and the oscillating signal of the photo detector were simultaneously measured, as shown in Figure 4(b).

The circles plot the measured current flowing through the quartz tuning fork and the thick solid curve shows the fitting of the current spectrum using the electric model in Figure 2(a). The dashed curve in Figure 4(b) shows the current produced by the mechanical oscillation of the tuning fork, and the thin solid curve is the oscillation amplitude of the tuning fork measured using the AFM. These latter two data sets allow an estimate of the coupling constant α which is found to be $10.4 \pm 0.1\ \mu$C/m.

3. RESULTS AND DISCUSSION

3.1. Measurement of the Interaction Force

Once the amplitude of the tuning fork is calibrated, it is very interesting to measure the tip-substrate interaction force versus separation. Briefly, when a force gradient $\partial F/\partial z$ acts on an oscillating tip, the resonant frequency will shift. As a first approximation, for small oscillation amplitude,[10] the interaction force can be related to the frequency shift by

$$\Delta\omega = -\frac{\omega_0}{2k}\frac{\partial F(z)}{\partial z} \tag{4}$$

where $F(z)$ is the tip-substrate interaction force, z is the closest separation between the tip and the substrate's surface while the tip is vibrating, k is the spring constant of the tuning fork, and ω_0 is the natural resonant frequency when the tip is far away from the substrate (i.e., when no interaction is present). Integrating Eq. (4), we can retrieve the interaction force. For large oscillation amplitude, the force can be retrieved using more complicated equations.[11,12]

To solve this problem for arbitrary oscillation amplitude, a general solution in terms of the frequency shift and oscillation amplitude was derived by Sader.[12] This important result is given by

$$F(z) = 2k\int_z^{\infty}\left(1+\frac{\sqrt{A}}{8\sqrt{\pi(\xi-z)}}\right)\Omega(\xi) - \frac{A^{3/2}}{\sqrt{2(\xi-z)}}\frac{d\Omega(\xi)}{d\xi}d\xi \tag{5}$$

Equation (5) forms the basis for our experimental extraction of the interaction force that act on the AFM tip when it is only a few nanometers away from the surface of the substrate.

3.2. Experimental Determination of Interaction Force Versus z

In this experiment, an AFM tip was mounted onto a tuning fork and inserted into an AFM head made by Nanotec Electronica. The tuning fork with a silicon probe attached is kept oscillating resonantly by a phase-locked loop (PLL) feedback. The resonant frequency shift (compared to the natural resonant frequency in the absence of tip-substrate interaction) and the amplitude of oscillation was measured while changing the tip-substrate separation in air room temperature. Typical data are shown in Figure 5(a). In this case, the substrate is freshly cleaved, highly oriented pyrolytic graphite which is electrically grounded. The tip is a sharp AFM silicon probe. From this data, the interaction force can be calculated using Eq. (5).

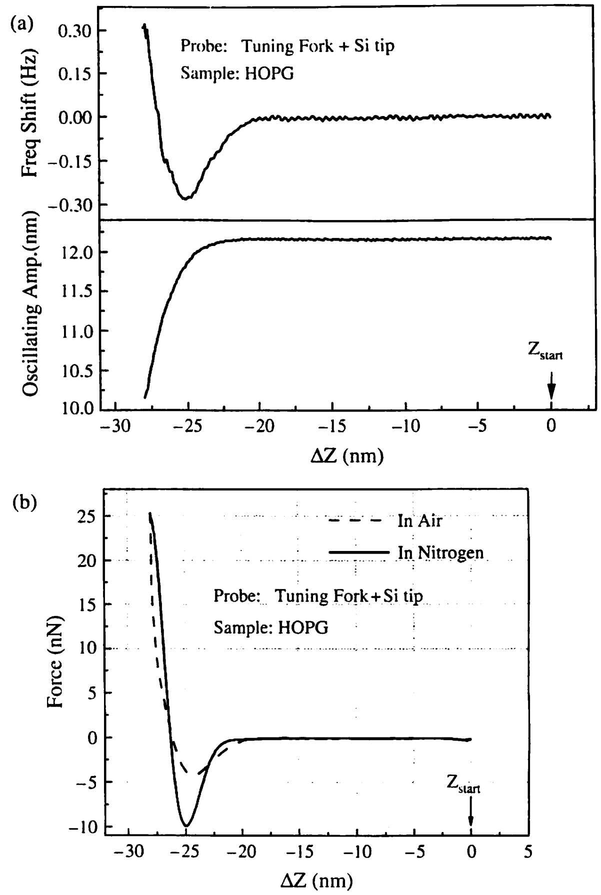

Fig. 5. (a) Oscillating amplitude (bottom) and resonant frequency shift (top) versus tip-substrate in air and at room temperature. (b) The calculated interaction force under different dry nitrogen and ambient air conditions.

Under ambient conditions, the silicon probe and substrate can be covered with a thin layer of water, which can impact the tip-substrate interaction force. To measure the influence of this thin layer of water, frequency versus z data has been acquired before and after dry nitrogen gas was used to backfill the chamber while keeping all other experimental conditions the same. The interaction force calculated from the frequency shift data is plotted in Figure 5(b). The data clearly shows a decrease in the interaction force from the dry to the ambient (~40% relative humidity) condition.

The rationale for performing this experiment is as follows. Prior SPM work suggests that no capillary water neck forms between a tip and substrate until a minimum thickness for an adsorbed water film is reached at roughly 40% relative humidity.[13] For a relative humidity above this value, the formation of a water neck between the tip and substrate would severely disrupt the frequency versus z data. For a relative humidity at or below this ~40% value, van der Waals interactions are expected to dominate.

With this background, a qualitative explanation for the observed decrease in the interaction force at ~40% relative humidity can be found by considering the influence of Hamaker constants on the interaction force between two dissimilar materials. For a sphere of radius R separated by a distance z from a flat plane ($z \ll R$), the interaction force is approximated by.[14]

$$F = -\frac{A_{12}R}{6z^2} \tag{6}$$

where A_{12} is inferred from the Hamaker constants of the sphere (A_{11}) and flat plane (A_{22}) using

$$A_{12} \approx \sqrt{A_{11}A_{22}} \tag{7}$$

For the system measured, we assume an SiO_2 tip surface when in a 0% relative humidity (dry nitrogen) environment, a hydrophobic HOPG substrate that does not adsorb water even when the relative humidity is greater than zero, and a thin water layer coating the SiO_2 tip under ambient (~40% relative humidity) conditions. As a first approximation, the relevant Hamaker constants for SiO_2, graphite and H_2O are 65 zJ, 275 zJ and 37 zJ, respectively.[15] Using these numbers, Equation (6) predicts the interaction force of a water coated SiO_2 tip with HOPG is reduced by ~33% when compared to a SiO_2 tip with no water layer. This result is in qualitative agreement with the results plotted in Figure 8 which shows a reduction in the interaction force by roughly a factor of 50%. The fact that the observed reduction in force is greater than that observed is tentatively attributed to the simplicity of the model outlined above.

4. CONCLUSION

As shown above, a quartz tuning fork is an ideal force sensor for measuring frequency shift and analyzing the tip-substrate interaction forces. With a high mechanical quality factor, it is able to sense frequency shifts of ~0.01 Hz. Furthermore, the high stiffness of a tuning fork prevents the tip from jumping into contact to the substrate. These advantages present an opportunity to systematically study the tip-substrate interaction force and surface potential between materials under a variety of different conditions.

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